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A Study of the Reproducibility of the Micro-Vac Technique As A Tool for the Assessment of Surface Contamination in Buildings with Asbestos-Containing Materials

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ABSTRACT: The Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations (D-5755-95) was balloted and passed in 1995. Estimates of the precision of this method was determined by examining historical data from a round robin laboratory study and as actual dust samples taken from buildings with asbestos fireproofing. The analysis followed the draft ASTM dust method that was in use at that time. Like most ASTM methods, the development of the D-5755-95 method involved a series of draft methods, each differing somewhat from its predecessor. While some changes were made to each draft, these changes were primarily made to the sample preparation and analysis sections of the method. No changes were made to the sections addressing sample collection. Since the dust sample data was generated using several draft methods and the final method, the differences between methods may have contributed to the data's variation. The first study consisted of dust samples that were sent to nine independent laboratories. The laboratories involved in the round robin study were provided with known weight amounts of asbestos contaminated dust that was collected from a building with in-place vermiculitic asbestos-containing fireproofing. Good correlation was found between laboratories when the number concentrations were normalized to the weight amount of the dust provided to the labs. The round robin study demonstrated good precision with an overall coefficient of variation (CV) of 0.71. Several laboratories demonstrated a CV in the range of 0.30 to 0.40. These low CV values showed a high degree of reproducibility for the analytical

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method and indicated little if any numerical contribution from large particles (≤ 2mm) that may have been in some samples.

A second study conducted from 1991 to 1996 examined a large database of dust samples that were collected from 38 buildings located throughout the United States. These buildings also contained a vermiculitic asbestos-containing fireproofing product. The samples were collected from surfaces beneath the fireproofing. No physical barriers or obstructions were present between the fireproofing and the surface sampled. Only buildings with at least three or more collected samples were included in the study. These sample sets were selected to control variation due to sample location, collection technique and type of asbestos-containing material (ACM) present. While samples collected within buildings did vary by more than one order of magnitude, statistical comparisons show a greater degree of variability between buildings than between samples collected within individual buildings. Additionally, this study showed good correlation with other similar data.

The two studies showed that asbestos surface dust measurements determined by the micro-vac technique are reproducible and may be used to determine asbestos surface concentrations in buildings during asbestos evaluations.

KEYWORDS: asbestos, analysis, dust, quantitative, round robin, buildings

Introduction

Analyzing surface dust for asbestos contamination has been of interest to consultants and industrial hygienists for some time. Early studies date back to at least 1935 when Huribut and Williams collected dust that had settled on rafters in a manufacturing plant *[1]*. They evaluated the asbestos content in the dust by polarized light microscopy (PLM) to help determine the industrial hygiene conditions in asbestos plants. Dr. Irving J. Selikoff suggested that the analysis of settled dust would help to evaluate secondary environmental exposures in homes from asbestos which was released from nearby factories *[2]*. Carter documented an early use of the microvac collection technique for asbestos surface contamination in 1970 *[3]*. Dust and fibers were vacuumed from work clothing using an air-sampling pump equipped with a filter cassette. The filters were analyzed by phase contrast microscopy (PCM) and reported as asbestos fibers per cm^2 of cloth. Asbestos consultants in the early 1980's began to collect information about the asbestos content of surface dust and debris. Various techniques were employed to collect and analyze these materials. Usually samples were gathered from surfaces by scraping or wiping up the dust and/or debris and analyzed by PLM *[4]*. This method worked well on the larger debris type samples. However, fine particulate dust samples typically would result in false negatives because PLM could not resolve most of the respirable-size asbestos structures found in dust. During this time frame the microvacuum method was beginning to be utilized to collect dust from building surfaces and most of these samples were analyzed by transmission electron microscopy (TEM) *[5]*. The TEM detected and identified even the smallest

asbestos fibers *[6,7]*. This more sophisticated method was slow to be utilized during the mid 1980's because of the low availability of TEM analysis and limited understanding of how to interpret the data that was generated. Greater interest in dust analysis was generated in 1989 after the USEPA developed a draft method for evaluating surface dust to assist them in their research. This method used the micro-vac technique for sample collection and TEM for sample analysis. This method was published after further refinement in October 1995 by the American Society for Testing and Materials (ASTM) as their D5755-95 Standard Method entitled, "Microvacuum Sampling and Indirect Analysis of Dust of Transmission Electron Microscopy for Asbestos Number Concentrations". The ASTM method employed the microvacuum sampling technique and the analysis of the collected dust by TEM and this has become the method of choice for most evaluations of asbestos contaminated dust. Most commercial TEM laboratories in this country currently provide this analysis for their clients.

A few individuals still question the method's value. They doubt the method's reproducibility and accuracy as well as its usefulness in determining what asbestos concentrations in dust may pose a risk to building occupants and workers *[8]*. These criticisms have been based primarily on the logic that since this method relies on an indirect preparation procedure and ultrasound for particle disbursement that complex asbestos structures must be disaggregated causing artificially high number counts *[9]*. To address some of these questions a study of the analytical method was performed and a review of a large database of dust sample results was conducted.

A round robin study was conducted in 1991 by Lee et al. and published in 1996 that reported that ASTM D 5755-95 method may have uncontrolled positive bias and large variability *[10]*. The study used three types of samples: (1) asbestos particles (0.5 to 1mm in size), (2) single large bundles of 7M chrysotile and (3) a laboratory mixture of asbestos and Arizona road dirt. However, both the selected asbestos particles and the 7M chrysotile bundles were only measured in two dimensions. The third dimension (height) of either the single particles or the chrysotile bundles was never measured and therefore it could not be determined if each of the laboratories in the study was receiving the same amount of material. Different size particles of asbestos would have a major impact on the numerical amount of asbestos present in the samples sent to those laboratories. Because of these problems each laboratory in the Lee round robin study could have received a different concentration of asbestos structures that would lead to significant variability for the test. Additionally, the Lee study used simulated building dust manufactured with asbestos and Arizona road dirt to expand their round robin study. Using this simulated building dust was more appealing than individual uncharacterized asbestos debris particles. However, this introduced variability into the study in the form of unknown collection efficiency of each sample and unknown starting concentration of asbestos structures. These unknowns could have factored large variabilities into the final results. Round robin studies have always required that the participating laboratories receive well-characterized samples to test laboratory differences. Crankshaw [11] documented that the micro-vac method provided good variability (<15%), precision and reproducible results when the starting asbestos dust material was well characterized with known mass concentrations of asbestos and non-asbestos materials.

The ASTM dust method went through five years of balloting before it was finally published in 1995. However, from 1991 to 1995, only minor changes were made to the actual analytical protocol. These changes may have contributed to the data's variation.

This paper will describe two studies that include inter-laboratory testing of a known weight of dust collected from vermiculite based fireproofing and a summarization of nearly 200 dust samples collected from buildings across the country that were analyzed by a draft of the ASTM D5755-95 method or by the final published method.

Experimental Methods

Study 1

A large volume of settled dust was collected during an asbestos evaluation of a high-rise office building located in the southeast. The dust was collected on top of an air distribution duct located directly below vermiculitic asbestos-containing fireproofing. The round robin study was performed using subsets of this dust sample. Before the dust sample was subdivided for the participating laboratories, it was sieved through a 2x2 mm fiberglass screen to remove any large particles. This study was initiated prior to the introduction of the 1 mm particulate screening into the sample preparation section of ASTM D5755-95 method. Therefore, the introduction of some particles greater than 1 mm, but smaller than 2 mm may have contributed to the data's variation. The sample was then mixed to assure homogeneity. Small portions of the sieved dust were placed in pre-weighed glass vials and adjusted to be in a similar weight range (8 to 12 mg each). The weighed dust portions were placed into new pre-weighed, clear plastic 37 mm air cassettes with a 0.8 μm pore size MCE filter. Each cassette was re-weighed to determine the final weight of the added dust. Laboratories were provided sets of two samples during each of the two rounds. Three laboratories participated in both rounds while the host laboratory analyzed seven of these prepared samples. The laboratories were requested to perform a dust analysis according to the proposed 1991 ASTM draft method using a 1/100 dilution and AHERA TEM counting rules *[12]*. Laboratory B reported only 3 of their 4 samples. Results were normalized to the number of asbestos structures per mg of dust. Results were evaluated to determine relative differences between the laboratories as well as differences within the individual laboratories.

Results

Study 1

The results obtained from the round robin, inter-laboratory study are summarized in Table 1. The results are reported in structures per mg of dust x 10^7 in Figure 1. The pooled arithmetic mean was 3.41 x 10^7 per mg with a standard deviation of 2.43 x 10^7 and a CV of 0.71. Results for the intra-laboratory comparison are shown

in Table 2. The mean results ranged from 0.92 to 5.97 x 10^7 structures per mg with a standard deviation range of 0.19 to 3.21 x 10^7, and a CV range of 0.12 to 0.76.

FIGURE 1

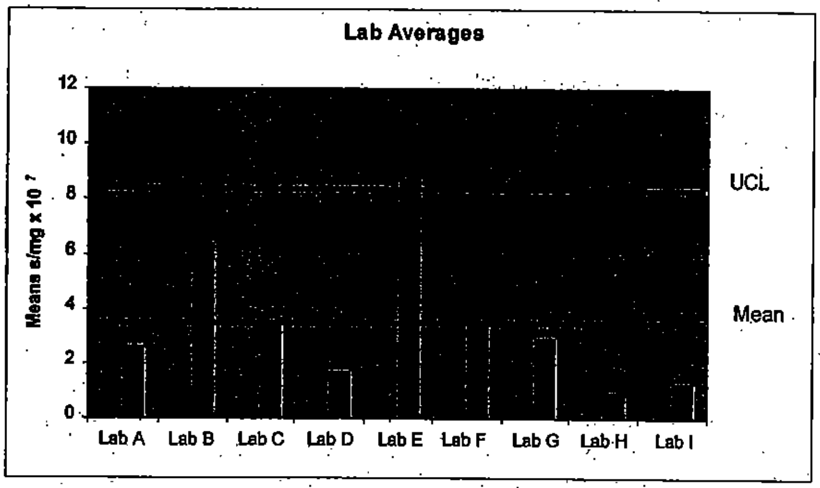

Table 1 - Inter-laboratory dust sampling analyses (asbestos structures / mg x 10^7)

Sample #	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	Lab H	Lab I
1	2.58	3.54	2.39	1.54	11	4.17	2.34	0.73	0.59
2	3.49	8.7	4.25	1.82	6.46	2.72	2.97	1.12	1.96
3	2.69	6.9					3.75		
4	1.67						1.88		
5							2.17		
6							3.11		
7							4.22		
Arithmetic Mean	2.61	6.38	3.32	1.68	8.73	3.45	2.92	0.92	1.28
Geometric Mean	2.52	5.97	3.19	1.67	8.43	3.37	2.81	0.90	1.08
Standard Deviation	0.75	2.62	1.32	0.20	3.21	1.03	0.86	0.28	0.97
CV	0.29	0.41	0.40	0.12	0.37	0.30	0.29	0.30	0.76
Pooled by Laboratory									
Arithmetic Mean	3.41								
Geometric Mean	2.75								
Standard Deviation	2.44								
CV	0.71								

These results show when laboratories receive a known amount of well homogenized asbestos-containing dust, the results show low variability for this type of analytical method as shown by the overall CV of 0.71 for all laboratories. This study is in contrast to the Lee inter-laboratory round robin study that used somewhat uncharacterized asbestos particles and Arizona road dust samples materials, but are in better agreement with the Crankshaw work at Research Triangle Park. However, if in Lee's study, individual laboratory CV's were calculated, most would have been between 0.30 and 1.00. The major variability in Lee's study is between laboratories with one laboratory clearly an outlier.

Discussion

Study One

One of the factors that may affect the variability of an inter-laboratory round robin study is the experience a particular laboratory may have performing the micro-vac dust analysis at that time. This inter-lab study was performed in 1991. During this time many laboratories may not have had much experience with the analysis of dust samples. This lack of experience is most likely the cause of the significant differences found between laboratories in this study as shown in Table 3. The significant differences detected between the laboratories is, however, primarily driven by high variability of measurements made by Lab B and particularly Lab E.

The two laboratories believed to have the most experience in analyzing dust samples by this method in 1991 were laboratories A and G. It was estimated that each of these two laboratories had analyzed in excess of 1000 dust samples by the time of this study. The results for laboratories A and G are nearly identical and each lab had a CV of 0.29.

Table 3 – *Anova: Single Factor*

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Lab A	4	10.43	2.61	0.555
Lab B	3	19.14	6.38	6.859
Lab C	2	6.64	3.32	1.730
Lab D	2	3.36	1.68	0.039
Lab E	2	17.46	8.73	10.306
Lab F	2	6.89	3.45	1.051
Lab G	7	20.44	2.92	0.733
Lab H	2	1.85	0.92	0.077
Lab I	2	2.55	1.28	0.937

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	114.801	8	14.350	7.192	0.000338	2.548
Within Groups	33.922	17	1.995			
Total	148.723	25				

Additionally, there have been some studies suggesting that the use of the ultrasonic water bath in the sample preparation would cause the uncontrolled breakdown of large (0.5 to 2.0 mm diameter) asbestos-containing particles in the dust sample, thereby producing anomalistic concentrations of asbestos structures [8,10]. Since the preparation of the test samples for this study included some particles as large as 2mm in diameter, following the theories proposed in the above cited papers, the expected inter-sample variations would be high. Each of the laboratories in this study used their own ultrasonic water bath for sample preparation. The make, model, and power setting of the bath was left up to the laboratory. If the ultrasonicator caused uncontrolled asbestos structure disruption, the coefficient of variation should have been much higher than the CV of 0.71 determined in this study. Also, the asbestos structure concentration range should have been found to be several orders of magnitude instead of the one order of magnitude observed. This study demonstrated low intra-lab variations and low overall variations. Therefore, there does not appear to be any appreciable contribution from randomly occurring particles as large as 2mm in diameter. The findings of this study are in agreement with an Environmental Protection Agency Study [13] which found that the indirect method does not cause asbestos structure breakup. Its main affect is to homogenize the particle suspension and provide a better particle distribution on the filter. This in turn provides for a more precise measurement of asbestos air and dust concentrations. To further improve the precision of the method, the current ASTM D5755-95 method instructs the sampler not to include particles greater than 1mm in diameter in the sampled area. The sample preparation section provides for the screening out of particles larger than 1 mm. Therefore, little concern should be made of this issue in the future.

Experimental Methods

Study 2

From 1991 to 1996, a large number of surface dust samples have been collected and evaluated. From this database of sample results, a subset of samples was selected from buildings which contained spray applied vermiculite based asbestos-containing fireproofing materials on structural components. Only buildings in which at least three or more samples were collected were included in this study. These samples were collected from areas either above the ceiling or in rooms with no ceilings or other obstructions which might interfere with the dust fallout from the fireproofing. The selected samples were collected primarily by one individual and were analyzed by the same laboratory in general accordance with a draft or the final version of ASTM D5755-95 method.

Results

Study 2

The results of 195 dust samples collected in 38 different buildings are summarized in Table 5. Table 6 displays the results of dust samples collected from six buildings that contain non-asbestos spray-applied fireproofing. Table 5 summarizes the 195 sample results collected from 38 buildings with spray-applied vermiculitic asbestos-containing fireproofing. The fireproofing contains approximately 10 to 12 percent chrysotile asbestos. These samples were collected from horizontal surfaces located in the ceiling plenum space or from mechanical or storage spaces that did not have ceilings or major obstructions between the fireproofing and the sample locations. Typically, these areas were dusty and frequently fireproofing debris was visible in nearby locations. While debris pieces (< 1 mm in diameter) were not collected, reasonable quantities of settled dust were easily collected from the surfaces. Since dust sample concentration data, like air concentration data, is most likely distributed in a log-normal manner, the geometric means along with the arithmetic means are presented.

A single factor Analysis of Variance (ANOVA) was performed on the data to determine if there were significant differences between buildings. The analysis of variance resulted in a significant F test indicating differences in asbestos dust concentrations between buildings.

While the ANOVA indicated that the data should not be combined, the summary in Table 5 was made to compare to other studies' results. It is also useful to determine an overall coefficient of variation (CV) for the method. The weighted geometric means for all 195 samples is 10.3 million s/cm^2 and the corresponding arithmetic mean is 15.8 million s/cm^2. The pooled standard deviation is 14.8 million s/cm^2 and the weighted CV is 0.97. This 0.97 CV included the recognized variations between buildings. The CV of 97% appears to be as good as if not better than the 100 to 150% EPA [14] estimated for direct analysis of air samples. This lower CV for dust analysis may be partly due to the indirect preparation of the samples, which provides for a more homogeneous distribution of the dust particles on the filter resulting in a more precise measurement.

TABLE 5- Summary of Dust Sample Results from 38 Buildings (structures/cm^2 x 10^6)

Building Number	No. of Samples	Geometric Mean	Arithmetic Mean	Standard Deviation	CV	Range
1	11	4.091	20.089	42.904	2.136	1.6 - 114.9
2	8	5.031	11.436	8.407	0.735	0.143 - 21.9
3	6	1.590	3.456	3.937	1.139	0.198 - 10.1
4	4	17.794	35.240	44.992	1.277	4.58 - 101
5	6	0.083	0.209	0.316	1.513	0.006 - 0.2
6	4	6.905	13.225	15.468	1.170	1.2 – 35.5

TABLE 5- Summary of Dust Sample Results from 38 Buildings (structures/cm^2 x 10^6) (Continued)

Building Number	No. of Samples	Geometric Mean	Arithmetic Mean	Standard Deviation	CV	Range
7	7	0.495	1.347	2.223	1.651	0.19 - 6.3
8	4	3.465	6.500	9.135	1.405	1.7 – 20.2
9	4	4.371	4.575	1.717	0.375	3.4 - 7.1
10	9	13.078	21.283	15.706	0.738	0.947 - 49
11	6	1.470	1.705	1.085	0.636	0.073 - 3.7
12	4	2.046	3.881	3.924	1.011	0.222 - 9.4
13	4	9.732	20.825	26.892	1.291	1.3 - 60.6
14	6	5.486	28.950	47.969	1.657	0.313 - 37.7
15	3	2.714	2.833	0.961	0.339	1.8 - 3.7
16	6	14.464	19.583	16.847	0.860	7.2 - 41.9
17	6	3.135	5.392	4.609	0.855	0.35 - 11.1
18	6	8.895	12.433	9.063	0.729	1.6 - 25.8
19	8	12.509	14.538	5.958	0.410	2.4 - 20.5
20	5	3.500	4.640	3.993	0.861	1.1 - 11.4
21	6	8.497	17.905	19.454	1.087	0.929 - 49.4
22	3	15.901	32.400	43.923	1.356	5.9 - 83.1
23	5	8.886	12.100	11.384	0.941	3.7 - 31.6
24	5	15.523	16.940	7.611	0.449	7.7 - 28.1
25	4	14.746	19.675	14.301	0.727	4.4 - 32.7
26	3	6.861	8.600	7.299	0.849	3.8 - 17
27	5	65.282	67.880	20.544	0.303	43.9 - 93.9
28	6	5.135	7.617	6.909	0.907	1.9 - 17.4
29	3	67.405	67.633	6.801	0.101	60.9 - 74.5
30	5	11.492	19.840	23.353	1.177	2.3 - 60.4
31	5	20.181	23.360	16.665	0.713	13.9 - 53
32	4	6.718	9.750	8.713	0.894	1.6 - 22
33	3	18.164	51.500	77.529	1.505	5 - 141
34	5	8.953	11.240	7.384	0.657	2.7 - 20.7
35	4	9.863	9.975	1.750	0.175	8.3 - 12.2
36	3	9.135	13.567	12.646	0.932	2.6 - 27.4
37	3	2.860	4.844	5.713	1.179	0.933 - 11.4
38	6	9.574	13.300	10.488	0.789	1.8 - 31.6
Buildings Total	Sample Total	Weighted Geo. Mean	Weighted Mean	Pooled Std. Dev.	Weighted CV	Overall Range
38	195	10.271	15.826	14.845	0.972	0.006 - 114.9

Table 6 summarizes dust sample data collected from 4 buildings that did not contain any major applications of asbestos-containing materials such as fireproofing. In

only 3 of 15 samples were any asbestos structures counted. In each case, only a single asbestos structure was counted. This is an insufficient count to conclude asbestos was present in the samples. These results demonstrate that the source of the asbestos contamination found in the dust from buildings with asbestos fireproofing is from the in-place ACM and not from some source outside the building.

TABLE 6 - *Dust Sample Results From Non-ACM Buildings*
Structures/cubic centimeter

Sample #	Building 1	Building 2	Building 3	Building 4
1	ND*	ND	ND	ND
2	ND	ND	ND	ND
3	ND		ND	ND
4	557		1300	
5	1300			
6	ND			

* Not Detected

Of the 35 field blank samples associated with 195 dust samples collected in all of the buildings, only 3 were found with 1 fiber counted and in 1 sample, two asbestos fibers were counted during the analysis.

Discussion and Conclusions

Study Two

The geometric mean of asbestos concentrations in the 38 buildings ranged from 83 thousand s/cm^2 to 67.4 million s/cm^2. The arithmetic mean ranged from 209 thousand s/cm^2 to 67.8 million s/cm^2. The lowest concentration on any of the surfaces was 6 thousand s/cm^2 and the highest concentration was 141 million s/cm^2.

In 1996 William Ewing and others [15] published similar data for surface dust samples collected in buildings with fireproofing. This data contained buildings with either vermiculite or mineral fiber based asbestos-containing fireproofing. The geometric mean of asbestos concentrations from 47 different buildings ranged from 17 thousand s/cm^2 to 52 million s/cm^2. The arithmetic mean ranging from 19 thousand s/cm^2 to 76 million s/cm^2. The lowest measured asbestos concentration was 1 thousand s/cm^2 and the highest was 220 million s/cm^2.

It appears that in both studies a similar range of asbestos concentrations and building average concentrations was found. All of the upper and lower ranges for

sample concentration and averages were in the same order of magnitude. The exception was the lower limit of the arithmetic mean that differed by one order of magnitude.

The range of asbestos concentrations found between buildings and, to a lesser degree within buildings, is due to a variety of factors. Certainly among these factors are the condition of the fireproofing material, past activities which have affected the fireproofing and any past cleaning of the surfaces which were sampled. Generally, dust collected in areas where asbestos-containing materials are in poor condition or where activities have impacted the materials will contain higher asbestos concentrations than dust collected in areas where materials appear in good condition and have little evidence of contact.

Maybe more important to an investigator than the absolute quantity of asbestos present in the dust is the location of the asbestos contaminated dust and its likelihood of disturbance. In many past studies the reentrainment of asbestos-laden dust from housekeeping, maintenance and renovation activities has been demonstrated. The resulting asbestos air concentrations from these reentrainment activities give rise for concern.

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